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Deterioration of Portland Cement Concrete.

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THE probable causes of cracks and disintegration of Portland cement concrete can be classified under (1) composition and physical state of the cement; (2) misuse of the cement, (3) composition and physical state of the aggregate, (4) conditions to which concrete is exposed, (5) poor workmanship.

Free Lime.

Poor quality cements have sometimes caused disintegration of concrete, particularly in reinforced concrete structures, due to the fact that these cements have expanded abnormally during the setting process. This expansion is generally caused by the presence of uncombined lime in the cement. Free lime is only present in a small amount in good Portland cement clinker, but its presence does not necessarily indicate that cement obtained from such clinker will be unsound. Thus if finely-ground calcium oxide is added to cement the mixture will not be unsound. The determining factors appear to be the fineness to which the CaO is ground and its physical condition, and the free lime should be in such a state as to permit it being hydrated before the cement attains its final set. Coarse grains of hard-burned lime hydrate slowly and cause expansion if present in appreciable quantity. Kühl¹ noted that expansion cracks in concrete are not due to the formation of amorphous Ca(OH)_2 but result from pressure caused by the growth of crystalline Ca(OH)_2 in one plane. Where the free lime is distributed evenly throughout the cement the growth of these crystals may cause the concrete to expand considerably in all directions, causing disintegration. Klein and Phillips² stated that the burning temperature and the fineness of grain are the controlling factors in determining whether the hydrated Ca(OH)_2 will be amorphous or crystalline. Lime subjected to a low burning temperature will hydrate rapidly, so that supersaturated solutions are quickly formed and Ca(OH)_2 is precipitated in the amorphous form. As the burning temperature is increased the CaO hydrates more slowly, so that a supersaturated solution is

only very slowly formed thus permitting deposition of $\text{Ca}(\text{OH})_2$ crystals. Expansion due to free lime may therefore be cured by (1) finer grinding, and (2) seasoning in a moist atmosphere. These processes permit of complete hydration of the free lime to amorphous $\text{Ca}(\text{OH})_2$ so that disruptive expansion does not occur in the Le Chatelier test. A cement which shows expansion cracks will not as a rule give such high strengths as would have resulted had it been sufficiently burned.

Copper Chloride and Lead Nitrate.

The presence of abnormal constituents in cement has also been found to have injurious effects upon its strength. The presence of Zn, Pb or Cu compounds in very small amounts considerably slows the set and reduces the early strength. Gonell³ showed that copper chloride and lead nitrate retarded crystal formation for seven days. Cements containing more than 0.02 per cent. of ZnO, PbO, or CuO should be rejected where high strength is required before seven days. According to Kühl⁴, additions of 2 per cent. of Mn, Zn, Cu, or Pb salts decrease the strength of Portland cement. Heyn⁵ showed that wet concrete attacks Pb, Cu, and Zn, particularly in the absence of carbon dioxide. In the presence of carbon dioxide a protective coating is formed over the metal.

Coal.

"Concrete and Cement Age" (Vol. iv, p. 62) states that lignite particles cause serious cracks in concrete and lower the compressive strength considerably. Anthracite particles show similar effects, though to a less extent.

Mixing Water.

Water used in mixing concrete should not contain large quantities of dissolved salts, as these tend to cause efflorescence. The water and the aggregate should be free from sulphides. Witt⁶ made both neat and sand briquettes of Portland cement using aqueous sodium sulphide of various concentrations (1 to 0.01 per cent.) as the gauging liquid, and showed that there is a considerable decrease in strength, approximately proportional to the concentration of sulphide solution used. The detrimental effect appeared to be most marked with cements high in iron. All the briquettes were badly discoloured.

Sugar.

Cases of deterioration, or in some instances failure to set, have occurred where either the cement or aggregate has become contaminated with sugar. Burchartz and Rodt⁷ found that a 0.1 per cent. sugar solution used in mixing gives improved results, but that higher concentrations are dangerous. They considered that such low concentrations do not merely produce their effect by solution of calcium hydroxide giving calcium saccharate, but that the sugar has an effect upon the colloidal actions concerned in the setting. Gonell⁸ gauged cement with 1 to 10 per cent. sugar solutions and stored the test pieces in air. Gel formation predominated at the expense of crystal formation, which was said to explain the occurrence of expansion and shrinkage as well as the slow

hardening. Crystal formation may not be evident until some months afterwards, but the addition of soda greatly increased crystal formation with precipitation of calcium carbonate. Calcium saccharate has an effect similar to sugar solution upon the setting process, so that the result is not due to the solubility of calcium hydroxide in sugar solution alone. Grün⁹ stated that formaldehyde, sugars, and similar organic compounds, even in small quantities, retard and almost completely prevent the hardening of cement.

Burchartz and Rodt⁷ found that sugar solutions produce complete disintegration of concrete even if the concrete has been allowed to harden before it is placed in contact with the solution. They ascribe this to the removal of hydrated lime to form calcium saccharate, thus rendering the concrete more porous and capable of progressive attack resulting in complete failure. Donath¹⁹ found that pieces of hard concrete immersed in 20 per cent. sugar solution deteriorated after three months. He found that the concrete lost calcium, aluminium, and iron and the sugar solution became coloured. Thus it seems inadvisable to use unlined Portland cement concrete for tanks for such liquors.

Carbon Dioxide.

The various agents which cause deterioration of concrete structures can be classified according to whether they occur in (1) the ground underlying the structure, (2) the liquid conducted or held by the structure, (3) the surrounding air.

Newman¹⁰ found that ground waters containing dissolved carbon dioxide are destructive to Portland cement concrete, owing to the leaching out of the lime as calcium bicarbonate. Freise²⁹ gives the following practical examples of the destructive action of waters containing dissolved carbon dioxide. In two mine galleries through limestone and slate and lined with 3 in. of concrete the original lining was damaged by the attack of underground water which carried 7.22 gr. of free carbonic acid per litre in addition to bicarbonates of soda and potash, lime, magnesia, and iron amounting to 0.363 gr. per litre, the mean temperature behind the lining being 22 deg. C. The quantity of lining destroyed in a year may be estimated at 25 cb. yd., or 1 cb. in. for each 29,000 cb. ft. of water. On an arch at the medicinal springs at Fervedouro, in the State of Minas Geraes, where the thermal waters contain 55 c.c. of free carbonic acid per litre of water (temperature 32 deg. C.) annual destruction has been recorded at the rate of 1 cb. in. per 166 cb. ft. of running water, no other harmful chemicals being present in the water. The intensity of action between free carbonic acid in underground waters and cement depends upon the speed with which the latter hardens; the more rapid the hardening the slower the subsequent action of the carbonic acid. Running or trickling water acts much more intensely than stagnant water. The following remedies have been suggested where concrete is exposed to water containing dissolved carbon dioxide. (1) Coat the exposed surface with two $\frac{1}{2}$ -in. layers of rapid-hardening Portland cement to secure an impervious surface free from pores. (2) Apply under pressure a coating of freshly-made silica gel to the still moist and fresh surface of the structure. Concrete

structures placed on marsh land or peaty soils are liable to deteriorate. Elliott¹¹ explained the rapid decay of Portland cement concrete drain tiles placed in peat soils as follows. The decomposition of peat results in the formation of organic acids which form calcium salts by reaction with the hydrated lime in the concrete. These salts are soluble in water containing carbon dioxide, so that when the water is running there is a steady leaching of lime from the tile. The formation of organic acids in marshy or peaty grounds or in water polluted with decaying vegetation is also mentioned by Freise²⁹ as being responsible for deterioration of concrete. Laboratory experiments made with concrete slabs demonstrated that lime and magnesia are the first constituents attacked by the humic acid solutions. Facilities for oxidation and salts such as carbonates, bicarbonates, and nitrates if present diminish the destructive action of the waters on concrete. Stagnant waters are less corrosive than rapid currents.

Lactic acid is developed by bacterial activity on the gummy constituents of decaying roots and stalks. Freise showed that lactic acid affects concrete even at a dilution of 1 in 40,000. In one test 320 days' action of a 1 in 5,000 solution of lactic acid in distilled water on a cement prism brought out 3.45 per cent. of the alumina content. The formation of lactates in crystalline form leads to increase of porosity and rapid access of the action to the interior layers. It seems that the disintegrating process is checked after a certain time by the formation of a protective crystallised film, but this only happens if the action of the acid does not reach the reinforcement. Freise examined the foundation of a concrete bridge in 1931. The upstream piles of the substructure were corroded to a depth of $1\frac{1}{4}$ in. on a length extending from 18 in. below the water level to about 3 ft. 2 in. deep. The reinforcement had been partially bared and corroded to 60 per cent. of its original diameter, and the concrete was deteriorated to such an extent that it could be easily removed with a pocket knife. It was found that when the bridge was erected the trees had only been cut down to water level, the roots being left. During 15 to 18 years the adjacent vegetation had produced at least 3,300 lb. of lactic acid (calculated on the lowest figures of growth, decay, and transformation by bacterial activity), which allowed an average concentration for the whole year of 1 in 500. Any organic sulphur compounds in the soil are oxidised to sulphuric acid which again leaches out lime. Dyckerhoff¹² stated that all concrete is more or less attacked by marsh water.

Where it is proposed to use Portland cement concrete tanks, containers, or pipes it should be remembered that in general all acids and salts capable of reacting with hydrated lime are liable to destroy the structure unless the product of interaction itself is non-destructive and therefore forms a protective coat for the inner structure.

Sewage and Gasworks Liquors.

Sulphide-containing liquors, such as sewage and gasworks liquors, are particularly liable to deteriorate concrete. The action in both cases is accelerated where there is ready access to air, causing the sulphide to be oxidised to sulphate. Weston¹³ observed and analysed concrete attacked by septic sewage and showed

that sulphuric acid is produced by the oxidation of hydrogen sulphide evolved from the decomposing sewage. This acid decomposes the cement, forming calcium and aluminium sulphates which do not withstand the action of the water. Most of the action occurs near the surface of the liquid and in confined spaces where the hydrogen sulphide tends to collect. Weston stated that the action would be diminished by a more thorough curing of the exposed surface and preventing the oxidation of the hydrogen sulphide by excluding the air or by providing sufficient ventilation to remove the gas. Dittol¹⁴ noted a case where concrete in a septic tank was attacked between the high and low water marks; this is another instance of oxidation under the influence of moisture and air. With regard to the action of gas-works liquors on concrete, Donath¹⁵ stated that the ammonium sulphide present converts some of the iron in the concrete into ferrous sulphide; ammonium carbonate also present dissolves out part of the iron. If the pipes or tanks are kept filled with the liquor to keep air away from the ferrous sulphide, concrete may be used with safety; but if the ferrous sulphide is oxidised to ferrous sulphate the cohesion of the concrete is diminished and it becomes pitted and will eventually fail.

Sulphite Liquor, Vinegar, Wine.

It is not advisable to store sulphite liquors, vinegar, or wine in unlined concrete since the free acids in these liquids attack the concrete; moreover the cement affects the freshness and bouquet of the wine.¹⁶ Brown¹⁷ stated that vinegar attacks a 1:2 cement mortar, leaving a soft dark coating which is easily detached.

Sulphates and Chlorides of Magnesium and Sodium.

Grün¹⁸ pointed out that concrete tanks cannot be used for storing solutions of sulphates and chlorides of magnesium and sodium unless great care is taken to obtain a structure of maximum density. Experimental tanks made of plain concrete were totally disintegrated by 10 per cent. solutions of these salts in one to two years. Freise¹⁹ points out that waters containing calcium sulphate are slowly harmful to Portland cement concrete; he considers that calcium sulphotoaluminate is formed together with an isomorphous crystalline mixture of gypsum with alum. Where concrete is to be placed in underground waters containing calcium sulphate it should be left to harden for some time before it is used; a solution of as low a concentration as 0.05 to 0.08 gr. per litre of calcium sulphate will delay the hardening process. Slow-setting cement will not harden in a solution of calcium sulphate of a concentration of 0.15 to 0.20 gr. per litre and should therefore not be used. Freise stored 2 m. by 2 m. by 3 m. prisms in calcium sulphate water; concentration varied from 0.21 to 1.85 gr. per litre of CaSO_4 ; temperature varied from 16 to 25 deg. C.; hardening period before immersion varied 3 to 60 days. The samples were left in solution from 5 to 10 months. A loss of strength ranging from 7.8 to 20 per cent. of the initial crushing strength resulted. Ammoniacal liquors should not be stored in Portland cement concrete tanks as they dissolve out hydrated lime with liberation of

ammonia. Contrary to the majority of investigators, Grün found that free ammonia does not appear to be harmful.

Petroleum, Mineral Oil, and Rape Oil.

Schumann²⁰ stated that the strength of cement is lowered to about the same extent by the action of petroleum, mineral oil, and rape oil where the cement had been previously hardened either in water or first in air and then in water. He found that the denser the mortar or concrete the better its ability to withstand the action of oils. Rape oil proved to have the most deleterious effect on mortars and concrete. He agreed with Gadd²¹ and other workers in finding that all oils and fats of animal or vegetable origin are destructive to concretes. The general explanation given is that these oils and fats consist essentially of glycerides of various acids, such as stearic, palmitic, and oleic, which are readily decomposed by all alkalis with the formation of a soap and the liberation of glycerine. Concrete in contact with these oils and fats is thus liable to deterioration owing to the formation of lime soaps. By chemical analysis of attacked concrete Gadd showed that as a result of immersing "green" concrete in cottonseed oil, 32 per cent. of the oil combined with the lime. He stated that "green" concrete is much more susceptible than old concrete because the calcium carbonate formed after exposure to the air in the latter does not take part in the saponification process. It is the calcium hydroxide of the set cement which is active in the formation of lime soaps. Gadd showed that cotton seed and colza oils are absolutely destructive to concrete, while Canevazzi²² in tests on two-year old 1 : 3 sand mortars found a considerable action when it was immersed in olive oil. Lard oil also attacks the surface of concrete.

Freise²³ mentions the use of concrete tanks for storing Brazilian vegetable oils, partly palm kernel and partly euphorbiaceæ oils of different kinds. It was found in some cases that the concrete was slowly destroyed. As Gadd²¹ found, the destruction is due to the fatty acid of the oil forming a lime soap. The process of oxidation of the fatty acids, in which they become rancid, accelerates the disintegration. These facts were verified in a series of tests in which the temperature and degree of acidity were varied. With the exception only of acetic and butyric acids the rate of attack on the concrete is approximately proportional to the molecular weight of the acid, other conditions being equal. These remarks apply to the acids of the general formula $C_nH_{2n}O_2$ and $C_nH_{2n-2}O_2$. Rizinolic acid ($C_{18}H_{34}O_2$) is an exception. A temperature of 22 deg. C. is sufficient to start the reaction and the rate of reaction increases as the temperature rises. Thus in collectors for oil which has been pressed in cylinder presses under temperatures sometimes reaching 70 deg. C. the influence on the cement is much more pronounced.

Presscakes and extraction residues, especially if these are given time to accumulate moisture, are very severe in their action on concrete due to the high acid content developed; this is especially true of oiticica oil and Brazilian tung oil. Seeds which are commonly stored in their shells are quickly deleterious to concrete on account of the ulmic acids developed in moist surround-

ings. Humates, carrying large amounts of water of crystallisation, are formed under the influence of time, temperature, and moisture, and ultimately the crystallisation pressure cracks the cement surface.

The crushing of seeds is always accompanied by the development of carbonic acid in an alcoholic fermentation stage which has its origin in the fermentable carbohydrates. Although the transformation of the lime of cement into insoluble calcium carbonate seems to be favourable for the cement surfaces exposed, the constant action of carbon dioxide, especially in moist surroundings, such as occur in oil factories, leads to the decomposition of the cement surfaces due to the formation of bicarbonates.

Phosphoric Acid.

Grün²³ showed that phosphoric acid in low concentration is exceedingly slow in its action on concrete structures. Its slow disintegrating power is due to the slow formation of soluble acid phosphate. Deterioration has also been traced in some cases to the action of lactic and tannic acids. Lactic acid from milk fermentation causes disintegration by leaching out lime to form calcium lactate, while tannic acid arising from some skin-curing processes destroys concrete by forming tannates.

Oils Mixed with Cement.

Cases of the failure of concrete structures are recorded throughout the literature in which the primary cause was the presence of certain oils mixed with the cement. The object of these additions was undoubtedly to reduce the water permeability of the structure, but it is now realised that there is great risk attached to the addition of these oils. Gadd showed that most mineral oils and greases weaken concrete without in any way decreasing its water permeability. Feret²⁴ pointed out that the addition of heavy mineral oil to cement mortars diminishes the compressive strength as well as the adhesive power of the mortar. Brown¹⁷ showed that neat briquettes gauged with a mixture of 2 per cent linseed or machine oil and 21 per cent. water do not have so high a tensile strength at 7 and 28 days as do those gauged with 23 per cent. of water alone. Linseed oil, being saponifiable, reduces the strength to a much greater degree than does machine oil consequent upon the formation in the former case of calcium linoleate. Taylor and Sanborn²⁵ made tests on concrete mixed with asphaltic oils. They used five different kinds of oil containing from 21 to 92 per cent. asphalt. The results showed (a) a retardation of set, (b) tensile and compressive strengths decrease with increase in the quantity of oil used, the compressive strengths being more affected, (c) permeability to water under pressure (10 to 60 lb. per square inch) is increased by the presence of oil.

Sodium Silicate.

Sodium silicate has also been used to decrease permeability by water, but Meyer²⁶ showed that this substance quickens the set and also reduces the strength.

Sulphuric Acid.

One of the commonest forms of acid action to which building material is subjected is that of sulphuric acid derived from the oxidation of sulphurous gases in the atmosphere of large towns. This action is much less marked on concrete, possibly due to the formation of a protective deposit of calcium sulphate which closes up the pores. Grün¹⁸ stated that sulphurous acid as such probably causes little harm, but in practice it is always oxidised to sulphuric acid which is injurious. Dunn²⁷ carried out a number of experiments to determine the effect of sewage and sewage gases on concrete constituted of good material. He concluded that the gases in solution in sewage and those expelled from it, arising from its decomposition, can act injuriously upon concrete, when the following conditions prevail: (1) a high degree of putrescence of the sewage, (2) a moistened surface which absorbs the putrid gases, and (3) the presence of a free air supply. In the absence of one or other of these factors little danger from erosion need be feared. The work of Weston in this connection has already been referred to.¹³

Protective Measures.

In addition to the injurious agents mentioned there are incidental effects such as electrolysis and mechanical vibration, although cases of failure caused in this way do not appear to be frequent. Methods of avoiding or reducing deleterious influences of substances on concrete have been given by Freise²⁸; his conclusions are embodied in the following summary from the literature.

(1) Protection must be sought in the first instance in careful construction rather than in the use of a protective cover on the exposed surfaces. The concrete should be as dense as possible, all the pores between the sand particles being filled with cement and all the interstices in the filling materials being filled with mortar. This result can be obtained by careful calculation and by good mixing and placing. The concrete should be kept wet as long as possible: so long as the forms remain unstripped the concrete should be kept wet from above and the forms from beneath. After the construction is finished the concrete should be allowed to stand for at least a fortnight in an atmosphere free from vapours and gases while hardening proceeds. The reinforcing elements should have a cover of at least 1 in. on all sides, and more at places where liquids and vapours with possible deleterious effects remain undisturbed during the working of plant.

(2) A preventive chemical compound may be incorporated in the mix to afford protection. Trass and pozzolana are the most common substances added in this class, although hydrated lime, barium carbonate, and waterglass may also be used.

(3) An insoluble organic compound may be incorporated in the mix to prevent porosity. Certain soaps occur in this class.

(4) Superficial impregnation of a resistant substance may be carried out to prevent microporosity. Such substances include magnesium silicofluoride, magnesium aluminium fluoride, tars, and asphalts. The best surface finish is a

well-applied compact cover of cement of the same brand as is used in the construction of the work to be protected. Where only very weak solutions of deleterious liquids or vapours are to be expected, a hot solution of silicate of soda applied under pressure is effective. A coating of zirconium oxide is highly effective, but it is too costly to be a practical proposition; the coating material is obtained by mixing with sodium silicate 40 per cent. of zirconium oxide of such fineness that it passes a 200-mesh sieve. For each square yard of surface 0.37 to 0.65 lb. of this material is sufficient to treble the life of the treated piece, and the coating is effective against strong solutions. The lining of tanks or reservoirs with one of the so-called acid-proof tiles is not recommended, unless the joints between the tiles are also acid-proof and impermeable. Floors and passages laid in cement can be sufficiently protected by a double or threefold coating of asphalt applied hot.

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The Effect of Magnesia on Tetracalcium Alumino-ferrite in Portland Cement. By H. E. Schweite and H. zur Strassen. *Zement*, p. 511, 1934.—X-ray investigations of the system $4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3 - \text{MgO}$ show that there is no occurrence of a quaternary compound, and only mixed crystals are found. Hansen and Brownmiller's diagram for this system must therefore be corrected. It has been shown that $\text{MgO} \cdot \text{Al}_2\text{O}_3$ cannot form in Portland cement clinker.

Heat and Material Balances for a Rotary Kiln.

Experimentally determined material and heat balances for a dry-process rotary Portland cement kiln are reported by Messrs. William N. Lacey (California Institute of Technology) and Hubert Woods (Riverside Cement Company) in a recent number of *Industrial and Engineering Chemistry*.

The kiln tested was producing clinker from a dry mix for which the raw materials were limestone and clay. Final grinding of the raw mix was carried out in tube mills. Screen analyses showed 84 per cent. passing a 200-mesh sieve. The general arrangement of the kiln system is shown in Fig. 1. The diameters given were measured inside the refractory bricks. The gases passing up the stack were led through an electrical precipitator for recovering the dust. Movement of the gases was produced by natural draught in the stack. The fuel was residuum from Dubbs' cracking units for petroleum treatment, and contained carbon and

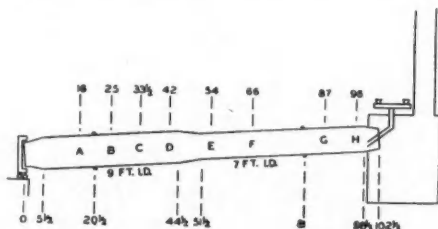


Fig. 1.—Kiln dimensions and thermocouple locations.

solid hydrocarbons in suspension. Its viscosity was very high at room temperature but decreased rapidly as its temperature increased. Special burners were used for this material, since ordinary fuel oil burners with small jets soon became clogged. The burners were of the type utilising air-atomising. Neither the primary nor secondary air used for combustion was preheated. The kiln was not equipped with a clinker cooler.

Temperature Measurements.

Temperatures of fuel entering the burner, kiln feed, and kiln room air were obtained with mercury-in-glass thermometers. The maximum temperature of the clinker in the kiln was measured with an optical pyrometer of the filament comparison type, by sighting through an opening in the kiln hood in such a way that observation through flames was avoided. The temperature of the clinker leaving the kiln was taken by sighting the optical pyrometer on the material just before it dropped over the lip of the kiln.

The temperature of the gases just leaving the feed end of the kiln was measured by a recording pyrometer with a chromel-alumel thermocouple in a metal protection tube. The couple and recorder were calibrated before use. A thermocouple

measuring the temperature of a gas stream is subject to a correction for radiation losses when the surrounding walls are at a lower temperature. In order to determine this correction, a comparison was made between the temperatures indicated by the regular couple and a thermally shielded couple, past which a rapid stream of the gases was drawn.

Since it was not feasible to operate the "high velocity" thermocouple for extended periods, a correction was obtained under usual operating conditions, and this was added to the temperatures obtained by use of the ordinary thermocouple. Under the conditions of this test the correction to be added was 450 deg. F.

In order to determine heat losses from the walls, sets of thermocouples were inserted at intervals along the length of the kiln. Each set consisted of three chromel-alumel couples made from No. 8 wires. The method of placing these couples is shown in Fig. 2. Since the two couples in the refractory brick were at different points in the same heat flow path they would give a measure of the

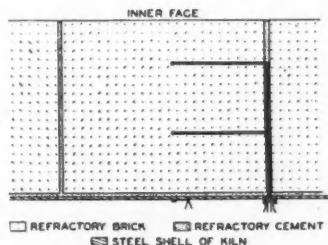


Fig. 2.—Arrangement of thermocouples in kiln walls.

temperature gradient through the brick if the distance between the two couples were known. From the temperature gradient and the thermal conductivity coefficient for the brick, the rate of heat loss through that portion of the wall was calculated. The third couple of each set was welded to the outside of the kiln shell and indicated the temperature of the exterior surface. The longitudinal positions of these thermocouple sets are shown by letters *A* to *H* in Fig. 1, the positions being given in feet from the lower end of the kiln. Readings were taken by attaching chromel and alumel leads to the respective wires of the couples. These leads ran to a thermos bottle filled with ice and water mixture, from which copper leads connected to the portable indicating-pyrometer instrument. Attaching the leads to the couple wires was accomplished by means of pinch clips to whose jaws the chromel or alumel lead wires were fastened in such a way that the lead wire was pressed firmly against the thermocouple wire of the same metal. This introduced no disturbing junctions at unknown temperatures. The readings from the three couples of a given set could be made in less than a quarter-revolution of the kiln.

Weighing Materials.

The fuel was weighed as it was fed to the burners by alternately filling, from the main fuel line, two open steel tanks which were provided with a manometer gauge indicating the pressure exerted by the liquid at the bottom of the tank. The weight of liquid in the tank corresponding to a given manometer reading was determined before the test by calibration against weighings and construction of a calibration curve. When one of the tanks had been filled with fuel, a weight measurement of its contents was taken. This tank then furnished the supply for the kiln, the fuel passing through a stream-driven pressure pump and a preheater on the way to the burners. When the liquid level had fallen near the bottom, the outlet valve was closed and the supply was then taken from the other tank, which had been filled and measured in the meantime. After closing the outlet valve of the first tank a measurement was taken to determine the weight of the residue before refilling. The difference between the initial weight and that of the residue gave the weight of fuel fed from the tank. In addition, weight readings were taken at one-hour intervals during the period of the test.

The clinker leaving the lower end of the kiln was weighed continuously in trucks before dumping into the regular plant clinker conveyor. The other stream of solid material leaving the kiln was in the form of dust carried out by the gases from the upper end. A portion of this dust deposited in the stack chamber is shown in Fig. 1. The rest was carried up the stack and collected by the electrical precipitators above. At regular intervals the dust collecting in the stack chamber was scraped out upon the kiln room floor and weighed. The electrodes of the treater were shaken down each hour and the total accumulation for the hour was weighed. This treater dust was very fine and thoroughly aerated and behaved very much like a fluid. This necessitated weighing in a bin mounted on a scale.

A direct measurement of the quantity of gases leaving the upper end of the kiln was found to be impracticable. The high temperature, the turbulence caused by the feed pipe, and the dust burden of the gases made Pitot tube readings taken at the end of the kiln uncertain. Dilution of the gases with air occurred upon entrance to the stack chamber and rendered subsequent gas measurement useless. It was therefore necessary to calculate the quantity of gases by indirect methods. Knowing the quantity and analysis of the fuel burned, the quantity of clinker produced, the analyses of kiln feed and clinker, and the analysis of the exit gases at the upper end of the kiln, it was possible to calculate the corresponding quantity of the exit gases.

Samples and Analyses.

The test continued for 48 hours. During this period both kiln feed and clinker were sampled four times each hour, each group of four being composited to give a representative hourly sample. The treater dust was sampled each hour at the time of weighing, while the stack chamber dust was sampled at the time of each cleaning-out and weighing. All these samples were subjected to "complete" analyses such as are ordinarily made in practice.

The fuel was sampled at the beginning and at the end of each 4-hour period,

the samples being composited to give a 24-hour sample. Upon these samples were made determinations of calorific value by the oxygen bomb calorimeter method, and of carbon, hydrogen, sulphur, nitrogen, oxygen, and ash by the usual methods of organic analysis. Since the analyses and measurement of the fuel were made upon a weight basis, there was no need to determine or use the specific gravity of the fuel.

The kiln gases were sampled by extending a sample tube into the upper end of the kiln, aspirating a rapid continuous stream of gas through it, and withdrawing slowly a small portion of this stream into a sample container by allowing the mercury originally filling the container to flow out through a capillary tube. The capillary was adjusted to require one hour for taking each sample. Gas samples were analysed by an improved Orsat apparatus, using mercury in the measuring buret. As the result of an extended investigation it was found to be impracticable to obtain a sample of gas which was representative of the entire stream. This was believed to be due to the high temperature at the upper end of the kiln. It could be observed that, under the conditions obtaining when operation was similar to that prevailing during the test period, the surface particles of the dry kiln feed glowed brightly at the upper end of the kiln, although the average temperature of the charge at this point was comparatively low. As a result of this condition, carbon dioxide was given off from the charge in substantial amount too near the point of sampling to permit thorough mixing of the gases before reaching the end of the kiln. As would be expected from this condition, the percentage of carbon dioxide in the sample varied with different locations of the sample tube over the cross section of the kiln. However, the ratio of oxygen to nitrogen (inert gases being called nitrogen here) appeared to be comparatively free of such variation for a given condition of kiln operation. This ratio, controlled by firing conditions at the lower end of the kiln and far enough away from the sampling point to ensure mixing, would be expected to remain unchanged by subsequent additions of carbon dioxide from the charge. This ratio of oxygen to nitrogen, together with the other measurements and analyses previously mentioned, furnished sufficient information to permit calculation of the average composition of the exit kiln gases and their quantity.

Data Obtained in Test.

The results described are average values for a representative 7-hour period of steady operation under one set of conditions.

The weight of fuel used was 18,445 lb. Its gross calorific value was 18,450 B.T.U. per pound. Its analysis (by weight) showed 86.2 per cent. carbon, 9.1 hydrogen, 1.2 sulphur, 2.1 oxygen, 1.2 nitrogen, and 0.2 per cent. ash. After deducting an amount of hydrogen necessary to give water with the oxygen remaining after allowance for enough to form sulphur dioxide, the "net" hydrogen was 9 per cent.

The average kiln gas analysis (by volume) for the period was 21.9 per cent. carbon dioxide, 1.8 oxygen, 0.1 carbon monoxide, and 76.2 per cent. nitrogen

(inert). As noted previously, the percentage of carbon dioxide cannot be depended upon accurately.

The measured weights and the percentage analyses of the solid materials entering and leaving the kiln are as follows :

	Kiln feed.	Clinker.	Treater dust.	Stack dust.
SiO ₂	14.7	22.3	11.8	12.0
Al ₂ O ₃	3.7	6.0	2.7	3.4
Fe ₂ O ₃	1.4	2.2	1.3	1.2
CaO	43.6	63.8	56.3	55.7
MgO	3.6	5.2	4.1	4.5
SO ₃	—	—	6.8	5.0
Loss	32.3	0.2	12.4	14.2
Total ..	99.3	99.7	95.4	96.0
Weight, lb. ..		79,670	4,830	820

Special analysis showed that the 32.3 per cent. loss on ignition of the kiln feed consisted of 29.1 per cent. carbon dioxide and 3.2 per cent. water. The moisture removable by drying at 221 deg. F. was 0.2 per cent. of the weight of the kiln feed. The low totals of the analyses of treater and stack dusts are largely due to the presence of alkali metals which were not determined.

The average temperatures for the 7-hour test period were as follows : kiln room air, 60 deg. F. (relative humidity, 50 per cent.) ; fuel, 178 deg. F. ; kiln feed, 176 deg. F. ; kiln charge (maximum), 2650 deg. F. ; clinker leaving kiln, 2440 deg. F. ; kiln gas at exit, 1950 deg. F. The temperature measurements from the thermocouples in the kiln walls gave the following average values (in deg. F.) ; the letters refer to the positions shown in Fig. 1 while the column headings refer to the thermocouples shown in Fig. 2 :

	Inner.	Outer.	Shell.		Inner.	Outer.	Shell.
A	1400	540	290	E	1460	610	355
B	1780	680	380	F	1370	670	360
C	1850	760	420	G	1060	480	310
D	1700	800	400	H	1140	560	—

Material Balance Calculations.

The weight of kiln feed, which was not measured, was calculated on the basis of a balance of total lime entering and leaving the kiln during the test period. The lime entering the kiln feed was set equal to the total lime in the clinker, the stack dust, and the treater dust. From the lime in the feed and the analysis of the latter its total weight was calculated. The value obtained was 123,600 lb. A similar balance using silica instead of calcium oxide gave 124,600 lb., and a third balance using all non-volatile constituents together gave 124,700 lb. The average of these three was 124,300 lb., which was taken as the weight of kiln feed.

In order to calculate the correct gas analysis it was assumed that the relative proportions of oxygen, carbon monoxide, and nitrogen found were correct. Taking

the composition of air, for the purposes of calculation, as 20.9 per cent. oxygen and 79.1 per cent. nitrogen by volume, and comparing this with the oxygen and nitrogen ratio in the kiln gases, it is seen that 0.0894 of the oxygen of the entering air was unused in combustion and 0.9106 was used. The fuel contained 1325 pound-atoms of carbon, and if burned completely would consume 1325 pound-moles of oxygen and produce 1325 pound-moles of carbon dioxide. While the 1325 pound-moles of oxygen were used to oxidise the carbon of the fuel, there would be required 412 pound-moles of oxygen to oxidise the corresponding "net" hydrogen, making a total of 1737 pound-moles of oxygen used from the air, if one neglects the small amount of oxygen thus allocated but unused by the 0.1 per cent. carbon monoxide found in the kiln gas analysis. By taking the ratio of carbon monoxide to unused oxygen in the flue gas analysis as correct, it was calculated that this carbon dioxide amounted to 9.5 pound-moles and this would lower the total oxygen used by approximately 5 pound-moles, leaving 1732 pound-moles. As found above, this used oxygen corresponds to only 0.9106 of the total entering, which would then be 1902 pound-moles. With this oxygen there came from the air 7199 pound-moles of nitrogen. By adding the carbon dioxide contributed to the gases by the kiln feed, 821 pound-moles, to that produced by combustion (after deduction for 10 pound-moles of carbon monoxide), the total present in the kiln gases was found to be 2136 pound-moles. The total quantity of dry kiln gases would then be $2136 + (0.0894)(1902) + 10 + 7199 = 9515$ pound-moles, and their corrected analysis would be 22.4 per cent. carbon dioxide, 1.8 oxygen, 0.1 carbon monoxide, and 75.7 per cent. nitrogen.

The water vapour carried by the kiln gases came from the air, from the kiln feed, and from combustion of the fuel. That from the air was calculated from the amount of dry air admitted, previously found, and the relative humidity value found in the kiln room air, giving 82 pound-moles. The water vapour from the kiln feed was 223 pound-moles and that resulting from combustion of the fuel was 824 pound-moles. Adding these three quantities, the total water vapour in the kiln gases was found to be 1129 pound-moles.

Converting the number of pound-moles of each constituent in the kiln gases to pounds and adding the values so obtained indicated 301,400 lb. of dry kiln gases and 20,300 lb. of water vapour, or a total weight of moist kiln gases of 321,700 lb. The weight of incoming moist air, similarly calculated, was 264,000 lb.

We are now in a position to strike a material balance. The following table shows such a balance between ingoing and outgoing streams of materials:

Ingoing Materials.			Outgoing Materials.		
	lb.	Per cent.		lb.	Per cent.
Fuel	18,445	4.5	Clinker	79,670	19.6
Kiln feed	124,300	30.6	Treater dust	4,830	1.2
Moist air	264,000	64.9	Stack dust	820	0.2
			Moist kiln gases	321,700	79.0
Total	406,700		Total	407,000	

It is interesting to note the large percentage of the ingoing materials contributed by air entering the lower end of the kiln and the even larger percentage which the kiln gases represent in the outgoing materials. The discrepancy of 300 lb. (< 0.1 per cent.) between ingoing and outgoing totals is fortuitously much smaller than the probable error.

Heat Balance Calculations.

For the purposes of heat balance calculations an arbitrary datum temperature of 60 deg. F. has been chosen. This was the temperature of the air in the kiln room, and for this reason the incoming air stream carried with it no sensible heat when considered from this datum. The sources of heat supplied to the kiln were the heat produced by combustion of the fuel, the sensible heat of the pre-heated fuel, and the sensible heat of the kiln feed. The heat of combustion of the fuel was obtained from the fuel weight and its gross calorific value, being equal to

TABLE I. HEAT BALANCE.

Heat supplied.	B.T.U.	Per cent.
Heat of combustion of fuel	340,300,000	98.9
Sensible heat of fuel	870,000	0.3
Sensible heat of kiln feed	2,880,000	0.8
Total	344,100,000	
<hr/>		
Heat used and lost.		
Heat used in reactions	64,600,000	18.8
Sensible heat of clinker	46,850,000	13.6
Sensible heat of kiln gases	166,500,000	48.4
Potential heat of combustion of CO ..	1,160,000	0.3
Latent heat of water from combustion	15,700,000	4.6
Heat lost through walls	28,650,000	8.3
Heat not accounted for	20,640,000	6.0
Total	344,100,000	

340,300,000 B.T.U. The sensible heat of the fuel was obtained from its weight, the difference between its temperature and datum temperature, and its specific heat which was taken as 0.40, the calculation giving 870,000 B.T.U. Consultation of International Critical Tables indicated the most probable value of the specific heat of the kiln feed between 60 deg. and 176 deg. F. to be 0.20 B.T.U. per pound per deg. F. Using this value, the sensible heat carried in by the kiln feed was found to be 2,880,000 B.T.U. Thus, the total heat input was 344,100,000 B.T.U. for the 7-hour period.

During the calcining and clinkering processes in the kiln there are several reactions involving heat effects. Dehydration of clay is an endothermic process. For clays cited by International Critical Tables of similar moisture content to that used in the kiln feed under consideration, the heat requirement for dehydration is 4000 B.T.U. per pound of water removed. Using this figure with the known water content of the kiln feed, the heat absorption for clay dehydration was 15,910,000 B.T.U.

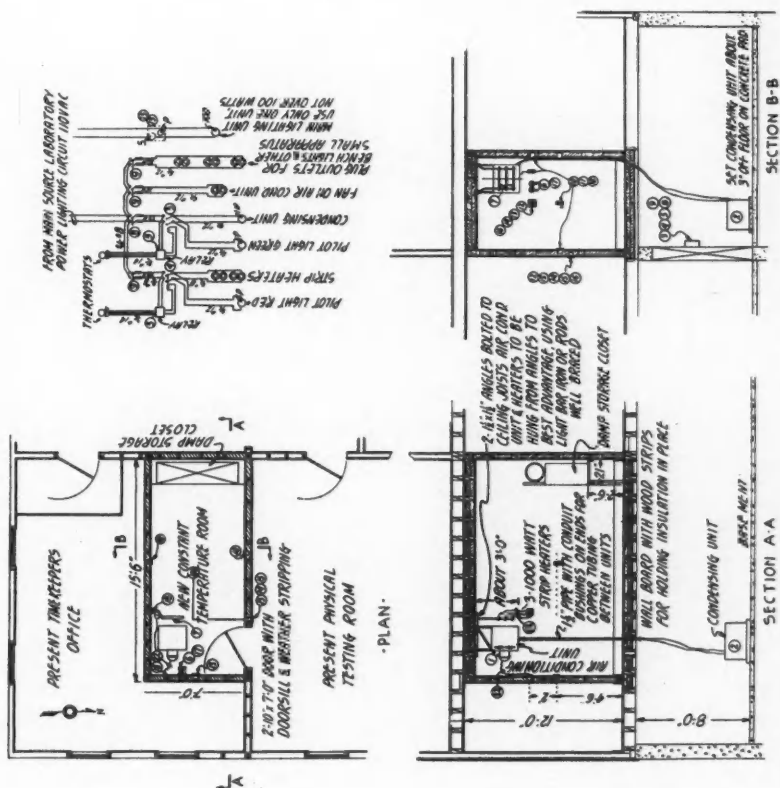
Heat is absorbed in decomposing calcium carbonate and magnesium carbonate, to form calcium oxide and magnesium oxide. In the present case it was not known how much of the carbon dioxide of the kiln feed was combined with calcium oxide and how much with magnesium oxide. It was therefore assumed that the number of moles of calcium carbonate and of magnesium carbonate were proportional to the moles of calcium oxide and magnesium oxide given in the kiln feed analysis. On the basis of this assumption and the known amount of carbon dioxide present, there were found to be 85 pound-moles of magnesium carbonate and 737 pound-moles of calcium carbonate. Using 50,000 B.T.U. per pound-mole as the heat of decomposition of magnesium carbonate and 76,500 B.T.U. per pound-mole for that of calcium carbonate, the heat required for decomposition of the carbonates was 60,640,000 B.T.U.

During the recombination of the oxides constituting the kiln charge to form the various cement compounds, an exothermic reaction occurs. This heat evolution amounts to approximately 150 B.T.U. per pound of clinker produced, or a total of 11,950,000 B.T.U. The net heat absorption for carrying on the various reactions in the kiln was $15,910,000 + 60,640,000 - 11,950,000 = 64,600,000$ B.T.U.

The hot clinker falling from the lower end of the kiln and the gas stream leaving the upper end of the kiln carry out heat, owing to their high temperatures. Harrison gives sensible heat curves for Portland cement clinker at various temperatures. From these data (the observed temperature and the weight of outgoing clinker) the sensible heat carried out in this material was 46,850,000 B.T.U. Using molal heat capacity equations corresponding to the different constituents of the kiln gases, their molal composition as previously calculated, the total quantity, and the exit temperature, the sensible heat of these moist exit gases was calculated to be 166,500,000 B.T.U. The carbon monoxide in the kiln gases represents a loss of potential heat of combustion which was calculated, from the quantity of this constituent present and a pound-molal heat of combustion of 122,400 B.T.U. to be 1,160,000 B.T.U. Since the gross heating value of the fuel was used, the outgoing kiln gases also carry out a quantity of latent heat corresponding to that obtainable by condensing at 60 deg. F. the water formed by the combustion process. This latent heat quantity was 15,700,000 B.T.U.

The remaining way in which heat could leave the kiln was by conduction through the walls. The amount of heat thus lost during the test period was estimated by determining the rate of heat loss at the thermocouple stations *A* to *H* (Fig. 1), using the temperature difference between the outer and inner couples in the refractory bricks, the distance between the junctions, and the thermal conductivity of the refractory materials, plotting from them a rate distribution curve for the length of the kiln to obtain a mean value and multiplying this value by the mean of the interior and exterior refractory surface areas and by the time. This calculation resulted in a value for the total loss of heat through the kiln walls of 28,650,000 B.T.U. The various amounts of heat entering and leaving the kiln are summarised in the heat balance in Table I. The large amount of heat lost in the high-temperature exit gases from this short kiln indicates the desirability of waste heat boilers for power development if such operating conditions as were followed during the test period were to be continued.

The new walls are of 2in. by 4in. studding, spaced at 16in. centres, with insulation between and lath and plaster on both sides. A new false ceiling was made by using 2in. by 8in. joists against the present plaster insulation, the lath and plaster being placed as shown. In the basement insulation was placed between



End section of moist closet.

the present joists and held in place with wall board. The insulation was carefully placed around all obstructions, so that there are no openings. The insulation used was rock wool in 15in. by 18in. by $\frac{3}{4}$ in. blocks, which was forced between the studding. These blocks can be cut to fit the odd-sized spaces. One layer of insulation was placed in all walls and two layers in the ceiling and under the floor.

The equipment and materials required (the number of the item corresponding with that shown on the drawings) were as follows: (1) air conditioner; (2) condenser; (3) and (4) two magnetic switches, 2-pole, 110-v.; (5) two Type A relays 110/20 v.; (6) two thermostats; (7) three strip heaters, 1,000 watts, 110 v.; (8) safety switch, 2 P.S.T., 60-amp., 250-v. fusible; (9) three safety switches, 2 P.S.T., 30-amp., 250-v., fusible; (10) six duplex receptacles, 3-wire polarised, flush; (11) six bakelite plates for receptacles; (12) two bakelite plates, 2-gang; (13) three pilot light receptacles, with 2 c.p., 125-v. lamps; (14) three bulls' eyes (1 green, 1 red, 1 clear); (15) toggle switch; (16) ten switch boxes (2in. by 3in. by 2in.; (17) six 4in. by 2 $\frac{1}{2}$ in. octagon outlet boxes; (18) four plugs, polarised; (19) cable, 2/c, No. 8; (20) cable, 2/c, No. 12; (21) cable, 2/c, No. 14 (or 12) BX; (22) Tirex (or equal) 3/c No. 14 (or 12); (23) eight connectors for 2/c, No. 8, BX cable; (24) fifty connectors, for 2/c No. 12, 3/c No. 14 cable.

The inside wood frame of the damp closet is made of dressed cypress, solidly constructed and fastened with brass angles and brass screws. The outside casing is No. 22 U. S. standard gauge copper, tinned on one side (the tinned side on the inside of the closet), with joints lapped and soldered. The outside casing is fastened to the wooden frame with round-head brass screws spaced about 4in. centre to centre, with solder around the screw heads. The sliding doors and door guides are of Monel metal No. 18 U. S. standard gauge. The door guides are fastened with round-head brass screws and soldered. Much condensation occurs on the inside of the casing and the construction must be such that this condensation stays on the inside of the closet. No temperature control is required for the moist closet as it is intended to be placed in the constant temperature room and is designed for ready transfer of heat into or out of the closet. The proper relative humidity is maintained by directing the fog nozzle into a rubber tube through which air is injected and mixed with the fog, thus humidifying the air and at the same time providing the necessary circulation of atmosphere throughout the damp closet. The water from condensed vapour dripping from the lower end of the rubber tube provides fresh water for the water storage tank below.

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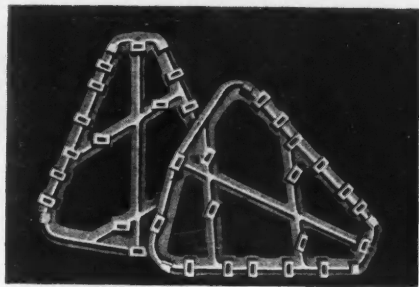
423,280.—Metallges. Akt.-Ges., 45, Bockenheimer Anlage, and Wendeborn, H., 103, Mithrasstrasse, both in Frankfurt-on-Main, Germany. Feb. 20, 1934.

Fine material, e.g., raw cement meal, is prepared for a sintering process by applying it as a coating to granular fragments of an inert substance of the same or similar composition as the sinter to be produced, for example, sinter returns. The granular core material is moistened and the fine material is then fed on to the moistened granules, for example, by dusting or strewing. The fine material may be applied in admixture with powdered fuel, or fine material and fuel may be applied successively. The moisture content of the finished material is preferably less than 20 per cent. and,

in the production of alumina cement or Portland cement, the mixture consists of 1 part of raw cement meal and 0.8 to 3 parts of sinter returns, the final moisture content being from 6 to 15 per cent. The water used for moistening the core material may contain substances which facilitate the adhesion of the particles of fine material, such as sulphite waste liquor, glue, metallic salts, etc. If desired the coated granules may be dried, for example by means of hot gases, before the roasting or sintering operation. In the form shown in Fig. 1 core material such as sinter returns is fed by a belt conveyer (7) into a chute (3) leading to a rotary drum (1) at the entry end of which is located a sprinkler (4) for moistening the core material.

The moistened material passes into a chute (5) where it is dusted with the fine

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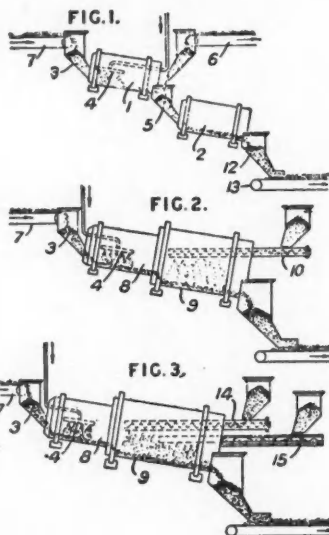
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material or fine material and fuel fed to the chute by a belt conveyer (6) and a hopper. The mixture then passes into a second rotary mixing drum (2) and then

(8) of smaller diameter in which the sprinkler (4) is situated and a larger portion (9) in which the strewing or dusting devices are situated. Alternatively two separate drums could be used instead of the two portions (8, 9) of a single drum. The strewing device may comprise a worm or vibro conveyer (10, Fig. 2), the casing of which preferably has a perforated bottom.

Fig. 3 shows an arrangement in which the fine material and fuel are strewn successively on to the moistened core granules, the fine material by the worm conveyer (14) and the fuel by the vibro conveyer (15), the whole or part of the bottoms of the conveyer casings situated inside the drum being preferably perforated. The depth of the charge of granulated material on the blast-furnace grate is preferably kept fairly small, e.g., less than 40 cm., and the charge may comprise two or more layers, the layers differing in the relative proportions of fine material, core material, and fuel.



to a hopper (12) and belt conveyer (13). In the forms shown in Figs. 2 and 3 a single drum is used, comprising a portion

Portland Cements.

423,043. Pontoppidan, C., 90, Vejlesøvej, Holte, Denmark. July 24, 1933.

In the manufacture of Portland cement the calcareous material is ground in a dry or substantially dry state, the clay is ground wet, water being added if necessary, and the two ground materials are admixed before burning.

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